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TITLE: Pentachlorothiophenylsorbate

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ABSTRACTED-PUB-NO: JP 69010774B

BASIC-ABSTRACT:

Pentachlorothiophenylsorbate (I):-

Agricultural chemical.

Sorboyl chloride (0.5 g.) was added to a soln. of pentachlorothiophenol (1 g.) in 3% aqs. NaOH (10cc) and the mixture stirred at 65-70 deg.C for 2 hrs. After filtering, the residue was washed with 10% aqs. NaOH dried and recrystallised from chloroform/EtOH mixt. to give pentachlorothiophenylsorbate (1.1g., m.p.114-115 deg.C).

DERWENT-CLASS: C00

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(54) Process of Manufacturing Pentachlorothiophenylsorbate

- (21) Application No.: S41-52912
- (22) Filing Date: August 12, 1966
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DETAILED DESCRIPTION OF THE INVENTION

The present invention relates to a process of manufacturing pentachlorothiophenylsorbate, a novel compound, by reacting alkali salts of pentachlorothiophenol with sorboyl halides.

Regarding materials used in this invention, pentachlorothiophenol (including its alkali salts) is known as an agricultural chemical particularly for effective control of blast/rotten neck (piricularia oryzae) or powdery mildew or the like. Sorbic acids, which are added to table salts, are known for their excellent anti-mold and preservation advantages and are the neutral or acid starting material used for producing sorboyl halides. The object of the present invention is to produce a novel compound having characteristics of both of the above ingredients, pentachlorothiophenol and sorboyl halides. More specifically, the present invention produces a novel compound, pentachlorothiophenyl sorbate by reacting alkali salts of pentachlorothiophenol and sorboyl halides. Example reactions are represented by the following methods: [(1)] in an appropriate solvent (e.g. benzene), an excess amount of alkali salts of pentachlorothiophenol is reacted with sorboyl halides; alternately [(2)], pentachlorothiophenol is dissolved into alkali aqueous solution to obtain alkali salts, followed by addition of sorboyl halides. Desirably, the solution mixture is stirred while being heated. Coarse crystals of pentachlorothiophenyl sorbate are obtained by removing solvent from filtrate for the former method or by filtration. The coarse product is then washed in caustic alkali solution and dried, followed by recrystallization in a chloroform-ethanol solvent. The product has a melting point of 114-115 °C, demonstrating its high purity, with a 60-90% yield. The pentachlorothiosorbate thus obtained by the present invention is valuable in that it finds useful application in agricultural chemicals and the like.

EXAMPLE 1

To a tri-neck flask, 1.5g of sodium salt of pentachlorothiophenol, 0.5g of sorboyl halide and 15cc of benzene (solvent) were added, followed by stirring. Continuous stirring of the flask in a hot bath while maintaining the temperature at 30 °C lets sodium salts of pentachlorothiophenol gradually disappear, and instead, precipitates NaCl. The color of the solution turned into greenish yellow after four hours. The solution was filtered to remove NaCl and un-reacted pentachlorothiophenol sodium salts. The filtrate was then heated in a hot bath at about 60 °C to remove benzene therefrom. The resulting crystals were washed in 10% NaOH solution and dried, followed by recrystallization in a chloroform-ethanol mixture solvent. 0.8g of pentachlorothiophenylsorbate crystals having a melting point of 114 ? 115 °C were obtained (at a 60% yield).

ANALYTICAL RESULTS (%)

	Cl	C	S	O	H
Theoretical Value	47.2	38.2	8.5	4.2	1.9
Actual Value	46.5	37.3	7.9	-	2.1

EXAMPLE 2

One gram of pentachlorothiophenol was dissolved in 10cc of 3% NaOH solution and 0.5g of sorboyl salts was added thereto. The solution initially looked like an emulsion; however, continuous

stirring of the solution at 65 ? 70 °C produced yellow precipitates. After 2 hours when sorboyl chloride no longer gave out a smell, the filtrate was filtered. [The filtrate] was washed in 10% NaOH and dried, followed by recrystallization in a chloroform-ethanol mixed solvent. 1.1 g ([yield] 90%) of pentachlorothiophenylsorbate crystals was obtained. Melting points and the results were the same as those obtained in Example 1.

WHAT IS CLAIMED IS:

A process of manufacturing pentachlorothiophenylsorbate by reacting alkali salts of pentachlorothiophenol with sorboyl halides.